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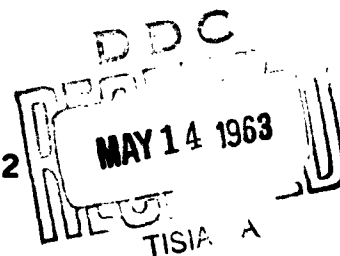
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THE DECOMPOSITION OF ARGENTIC AND
ARGENTOUS OXIDES IN CONCENTRATED
KOH ELECTROLYTE

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UNITED STATES NAVAL ORDNANCE LABORATORY, WHITE OAK, MARYLAND

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THE DECOMPOSITION OF ARGENTIC AND ARGENTOUS OXIDES
IN CONCENTRATED KOH ELECTROLYTE

Prepared by:
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and G. L. Cohen

ABSTRACT: The rate of decomposition of AgO in 40% (10N) KOH electrolyte was studied at temperatures ranging from 4.4°C to 71°C. No decomposition was observed at 4.4°C. After 35 days at 71°C there was complete reduction to Ag₂O and metallic silver. The addition of 6.5 - 8.5% ZnO by weight decreases the rate of decomposition of AgO and possibly prevents the decomposition of Ag₂O. The decomposition of Ag₂O to metallic silver under the conditions studied is thermodynamically feasible.

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Approved by: A. G. HELLFRITZSCH, Chief
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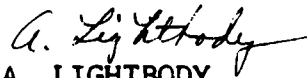
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This report covers the initial results of an investigation on the effects of temperature on the decomposition of silver oxides in concentrated potassium hydroxide electrolyte. The investigation has been performed under Task RUTO 3E000/217.

The results should be of interest to manufacturers and users of silver oxide-zinc batteries.

R. E. ODENING
Captain, USN
Commander


A. LIGHTBODY
By direction

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INTRODUCTION

The following two reactions



could affect the charge retention of the cathode in the AgO/KOH/Zn system. It is important, therefore, to know the rates at which the decomposition proceeds under various conditions. Measuring the volume of gas as it is evolved during the course of the above reactions is a convenient method for determining the decomposition rate.

To eliminate the unknown effects of organic separators, electrolyte additives, electrolyte concentration, and anode and cathode manufacturing processes, which are peculiar to the various silver-zinc cell manufacturers, the initial phases of this investigation were conducted with commercial chemically prepared AgO powder (approximately 99% AgO) in 40% (10N) KOH with and without ZnO additive. The experiments were planned so that not only the volume of gas would be measured but the silver oxide would be analyzed at the beginning and end to determine the amount of AgO converted to Ag₂O and Ag.

EXPERIMENTAL

The apparatus used was a gasometer designed by Otto and Eickel¹ which measures the gas produced by a chemical reaction (Fig. 1). As the sample in the container (5) gasses, the pressure forces the mercury in the manometer (11) away from the platinum wire (3) opening a light bulb circuit between contacts (12) and (13). When mercury is removed from the reservoir (6) through stopcock (7), the mercury in the manometer (11) rises closing the light bulb circuit. The amount of gas evolved from the sample can be calculated from the amount of mercury removed. The accuracy of the results is dependent upon a gas tight system and accurate temperature control. The leakage of the apparatus was tested by a mass spectrometer and found to be less than 2 cc/yr.

Table I shows the conditions under which the experiments were performed. The reactants were placed in constant temperature boxes (ovens or refrigerators). Necessary precautions were taken to ensure that the mercury was removed from the

system when it was at the temperature of study. Two forms of AgO supplied by Handy and Harman were used for these experiments. One was the type which purportedly Amlie and Ruetschi² studied. The second, labeled "low gassing" by the manufacturer, was reportedly the result of improved process control. Both forms were analyzed for total oxygen content by heating to 500°C to drive off the oxygen and for silver by the Volhard³ method. These analyses indicated that both materials were more than 99% pure AgO. However, analysis by the Topf and Diehl method as modified by Jirsa⁴ indicated a purity of only about 95%. Analysis by x-ray diffraction showed both alpha and beta forms of AgO and a trace amount of Ag₂O. No free silver was detected in either material.

To determine the actual composition of the gas evolved, a sample was removed from the gasometer and analyzed by gas chromatography. Since this analysis showed a definite increase in the order of 5% in the proportion of oxygen over that ordinarily contained in air, and no increase in the proportion of nitrogen, hydrogen or carbon dioxide, it was concluded that only oxygen was evolved.

RESULTS AND DISCUSSION

Figure 2 shows a comparison of the rates of decomposition of AgO in 10N KOH at three of the four temperatures at which the studies were made. These data show that the rate of oxygen evolution increases with increasing temperature. At 4.4°C (40°F) no evolution of oxygen was observed during a 70-day period.

At 71°C (160°F) the total volume of O₂ evolved after 35 days is 7 ml (STP) greater than the amount of O₂ (45 ml STP) which should have been evolved if only reaction (1) had taken place and gone to completion. At this point the gas evolution had apparently ceased, and the sample in the gasometer consisted of a white spongy material on top of a black powder. The two layers were physically separated and analyzed by x-ray diffraction. The analysis (Fig. 3) of the black layer showed that this material was principally Ag₂O with small amounts of Ag⁰ and Ag₂CO₃. Analysis (Fig. 4) of the white spongy layer showed that this material was principally Ag⁰ with small amounts of Ag₂O and Ag₂CO₃. No AgO was found in either layer. Thus, at 71°C, in the presence of 10N KOH electrolyte, silver oxide is reduced to metallic silver.

Figure 5 shows the degree of reproducibility when data were taken by two of the authors of this paper independently. There is a 10% deviation after eight weeks. This may be due to

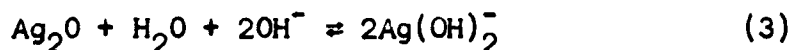
variation in temperature control at the time of obtaining data.

Zinc oxide dissolved in KOH reduces the rate of decomposition. Figure 2 shows the comparison of the decomposition of AgO in 10N KOH with and without ZnO at three of the temperatures studied. The results obtained with ZnO present are in agreement with Amlie and Ruetschi.² After 50 days the total volume of oxygen evolved was 48 ml. Gassing had apparently ceased. Experimental error as shown by Fig. 5 is about 10%. Assuming the experimental variation, the 48 ml value indicates that in presence of zinc oxide the decomposition proceeds only according to reaction (1). X-ray diffraction studies showed that the residue did not contain any metallic silver.

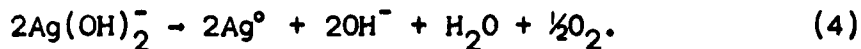
At 32.2°C (90°F) and 47.5°C (118°F) the presence of ZnO also reduces the rate of oxygen evolution. These results are consistent with those reported by Wylie.⁵ He also reports that Handy and Harman chemically prepared AgO had higher gassing rates than other AgO materials studied. He states that the gassing of various AgO materials is dependent upon the surface area of the samples used. Since no determination of surface area was made here, a comparison is not possible.

The reduction of silver oxide to silver in the presence of strong KOH electrolytes has not been reported until recently. Farmery and Smith⁶ attribute the phenomenon to the presence of some reducing impurity in KOH. However, the reduction of Ag₂O to Ag is thermodynamically feasible if Ag₂O dissolves as a negative ion of the form Ag(OH)_n⁻. Dirkse⁷ suggests Ag(OH)₂⁻ as the ionic species.

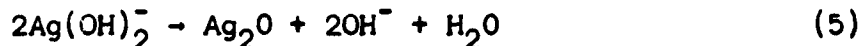
Assuming the solution reaction



then the reaction for reduction to metallic silver can be written as



For a study of thermodynamic feasibility, this reaction can be broken up into the following three reactions:



The value of the Gibbs standard free energy at 25°C (298°K) (ΔG_{298}°) for reaction (4) can be calculated by using the values of ΔG_{298}° for reactions (5), (6), and (7).

The solubility of Ag_2O in 10N KOH at 25°C has been reported to be 4.16×10^{-4} moles per liter.² The equilibrium constant for reaction (3) can be written as

$$k = [\text{Ag}(\text{OH})_2^-]^2 / (\alpha_{\text{OH}^-})^2 \quad (8)$$

and ΔG° can be calculated from the formula

$$\Delta G^\circ = -RT \ln k. \quad (9)$$

The concentration of KOH in the systems studied was about 11.89 molal. However, the precise activity of OH^- ion (α_{OH^-}) is unknown. Calculations from the data of Akerlof and Bender⁸ indicate that the activity coefficient (γ) is 9.66, and the activity, therefore, is 114.8. The value of free energy calculated for reaction (3) is $\Delta G^\circ = 14.6705$ kcal/mole. This value is the negative of that for reaction (5).

From values of E° for reactions (6) and (7),⁹ by means of Eq. (10)

$$-\Delta G^\circ = nfE^\circ, \quad (10)$$

where f is the Faraday constant, values of ΔG° for the reactions were calculated. These values, along with ΔG° for reaction (5), are tabulated below.

<u>Reaction</u>	<u>ΔG°</u>	<u>E°</u>
(5)	-14.671	
(6)	-15.775 kcal/mole	.344
(7)	18.451 kcal/mole	-.401
(4)	-11.995	

$$\text{Now} \quad \Delta G = \Delta G^\circ + RT \ln k, \quad (11)$$

where k for reaction (4) is

$$k = (\alpha_{\text{OH}^-})^2 P_{\text{O}_2}^{1/2} / [\text{Ag}(\text{OH})_2^-]^2. \quad (12)$$

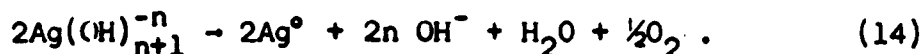
Using previously stated values for α_{OH^-} and concentration

of $\text{Ag}(\text{OH})_2^-$, one can calculate that ΔG will be negative for any value of P_{O_2} less than about 10^8 atm. Therefore, if reaction (3) is a correct representation of the solubilization of Ag_2O in KOH , the reduction to metallic Ag^0 is spontaneous, assuming $\text{Ag}(\text{OH})_2^-$ is the starting material.

It can be further shown that if the dissolved species is not $\text{Ag}(\text{OH})_2^-$ but $\text{Ag}(\text{OH})_{n+1}^{n-}$, then the reduction will still be spontaneous. In general, one can write reaction (11) for solution of Ag_2O . When this reaction,



is reversed and added to reactions (6) and (7), the overall reduction reaction is



The equilibrium equation for reaction (13) is

$$k = [\text{Ag}(\text{OH})_{n+1}^{n-}]^2 / (\alpha_{\text{OH}^-})^{2n}. \quad (15)$$

Since $\alpha_{\text{OH}^-} = 114.8$ in 40% KOH solutions, as n increases, k will decrease, $\ln k$ will become more negative, and ΔG° for reaction (13) as calculated from Eq. (9) will increase in a positive sense. Therefore, ΔG° for reverse (13) will increase in a negative sense, making ΔG° for reaction (14) always negative and indicating that the reduction of Ag^+ to Ag^0 in basic solution is spontaneous.

Since Ag_2O is photosensitive, the possibility that light caused the decomposition was considered. However, since the sample was in a dark oven most of the time, this possibility was discounted.

It is probable that the reduction to metallic silver occurs in two overall steps, which can be represented by reactions (1) and (2). Thus Ag_2O is reduced to metallic silver. To verify this a sample of pure Ag_2O was placed in 40% KOH solution at 71°C . Within five days a substantial quantity of a metallic substance was observed on top of the dark Ag_2O layer.

To verify that ZnO prevents or retards the reduction of Ag_2O to metallic silver, a sample of Ag_2O was placed in KOH and ZnO solution. Whereas the presence of silver was first observed within five days without ZnO in solution, no silver was observed after 20 days in the solution containing ZnO .

In Fig. 6 the rate of thermal decomposition for "low gassing" AgO is compared with that for ordinary AgO. Although the total gas evolved per gm AgO was only 32 ml, a partially metallic residue was observed. X-ray diffraction showed that this material contained metallic silver.

Figure 7 shows the rate of gassing of a section of a Yardney Silvercel cathode in KOH solution compared with the rated gassing of AgO. There is less oxygen evolved from the cell electrode, which is expected since the AgO powder has a larger surface area than the pressed electrode.

Figure 8 illustrates the increase in volume of evolved oxygen with temperature after four periods of time.

ANALYSIS PROCEDURES

To correlate the gas evolution data with the composition of the silver residues, a search for an accurate quantitative analysis was initiated. Various chemical methods as well as x-ray diffraction were investigated. X-ray diffraction is a useful tool for rough quantitative work but does not have the required accuracy for this project. There are two main problems involved in a chemical determination. The first problem is finding an analytical method suitable for the determination of the % AgO, % Ag₂O and % Ag in the same sample. The second problem is finding a suitable wash solution which will remove the KOH and ZnO without changing the composition of the residue. Alternatively, one might attempt an analysis in the presence of KOH and ZnO.

It was found that the major portion of the KOH could be removed from the residue by filtration through a sintered glass crucible. However, the small amount remaining after such a process is enough to cause significant error in the analytical results. Water is not suitable for use as a wash solution because the residues were found to be somewhat soluble. Water which had been left in contact with AgO and Ag₂O for a two-month period and then filtered was tried as a wash solution. Analysis of this solution before and after use indicated that the concentration of silver ion increased due to the washing process. Various alcohols were tried. However, it was found that in the presence of KOH a reduction of one or both of the silver oxides to silver occurred on the addition of alcohol.

None of the ordinary analytical procedures are suitable for the determination of the residue composition. By the Volhard method³ only the total per cent Ag in the sample can be determined. Whether the silver is present in the divalent,

monovalent or ground state cannot be determined by the use of this method. The Topf and Diehl method⁴ is suitable for determining the per cent AgO present in a sample but the presence of Ag₂O interferes with the determination.

A method of analysis whereby one could determine the exact composition of a sample containing AgO, Ag₂O and free Ag has been proposed by NAD Crane, Quality Evaluation Laboratory.¹¹ Through differential thermal analysis they determined that dry AgO decomposes on heating by a two-step process consisting of reactions (1) and (2). Their results indicated that each of the steps occurs at a distinct temperature: step (1) at 210°C and step (2) at 350°C or above. The 140°C separation between the steps of thermal degradation led them to propose the so-called "crucible method of analysis." This method essentially involves the following:

A sample, approximately 0.5 gm, is heated in an oven at 210°C for a three-hour period or until no further weight loss occurs. The weight loss is then determined and from this and the initial weight of the sample the per cent AgO present in the sample is calculated. The same sample is then heated at 450°C for a period of one hour and the second weight loss is determined. By subtracting weight loss #1 from weight loss #2 the weight loss due to Ag₂O originally present in the sample is determined and from this the per cent Ag₂O.

RESULTS AND DISCUSSION OF ANALYSIS PROCEDURES

To test the accuracy of the Crane method, samples of the same Handy and Harman AgO used in the gasometer determinations were analyzed. The results obtained, some of which are shown in Table II, consistently indicated an AgO-Ag₂O composition of greater than 100%. Since x-ray diffraction indicated only a trace of Ag₂O, it is probable that the large amounts indicated by the crucible method are in error.

The conditions used for the three pairs of samples in Table II were identical with respect to the AgO sample, equipment and temperature; however, the time of heating for step (1) was varied. The first pair was heated for three hours and the second and third pairs for four hours. The wide variation in results in samples heated for different times demonstrates that this method lacks precision and accuracy. The possibility that the heating period for step (1) of the decomposition was not long enough was investigated. However, when the material was maintained at 210°C for a period longer than four hours, partial decomposition to free silver occurred. A thermogravimetric analysis of the original Handy and Harman material was made

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using the Crane method. The results of this analysis are shown in Fig. 9 and show that the steps of the decomposition overlap one another. These results demonstrate that this method is not suitable for the determination of AgO, Ag₂O and Ag in the same sample.

CONCLUSIONS

- a. The rate of decomposition of AgO in strong KOH electrolyte increases with increasing temperature.
- b. In strong KOH electrolyte AgO will completely decompose to Ag₂O at 71°C after 35 days, and some Ag₂O will be reduced to metallic silver.
- c. The addition of ZnO to strong KOH electrolyte reduces the rate of decomposition of AgO to Ag₂O and prevents the reduction of Ag₂O to metallic silver.
- d. Free energy calculations indicate that the reduction of Ag₂O to metallic silver in 10N KOH is thermodynamically spontaneous, assuming a soluble species $\text{Ag}(\text{OH})_2^-$.
- e. A satisfactory method for the chemical analysis of mixtures of Ag, Ag₂O and AgO in the presence of strong KOH electrolyte has not been developed up to this time.

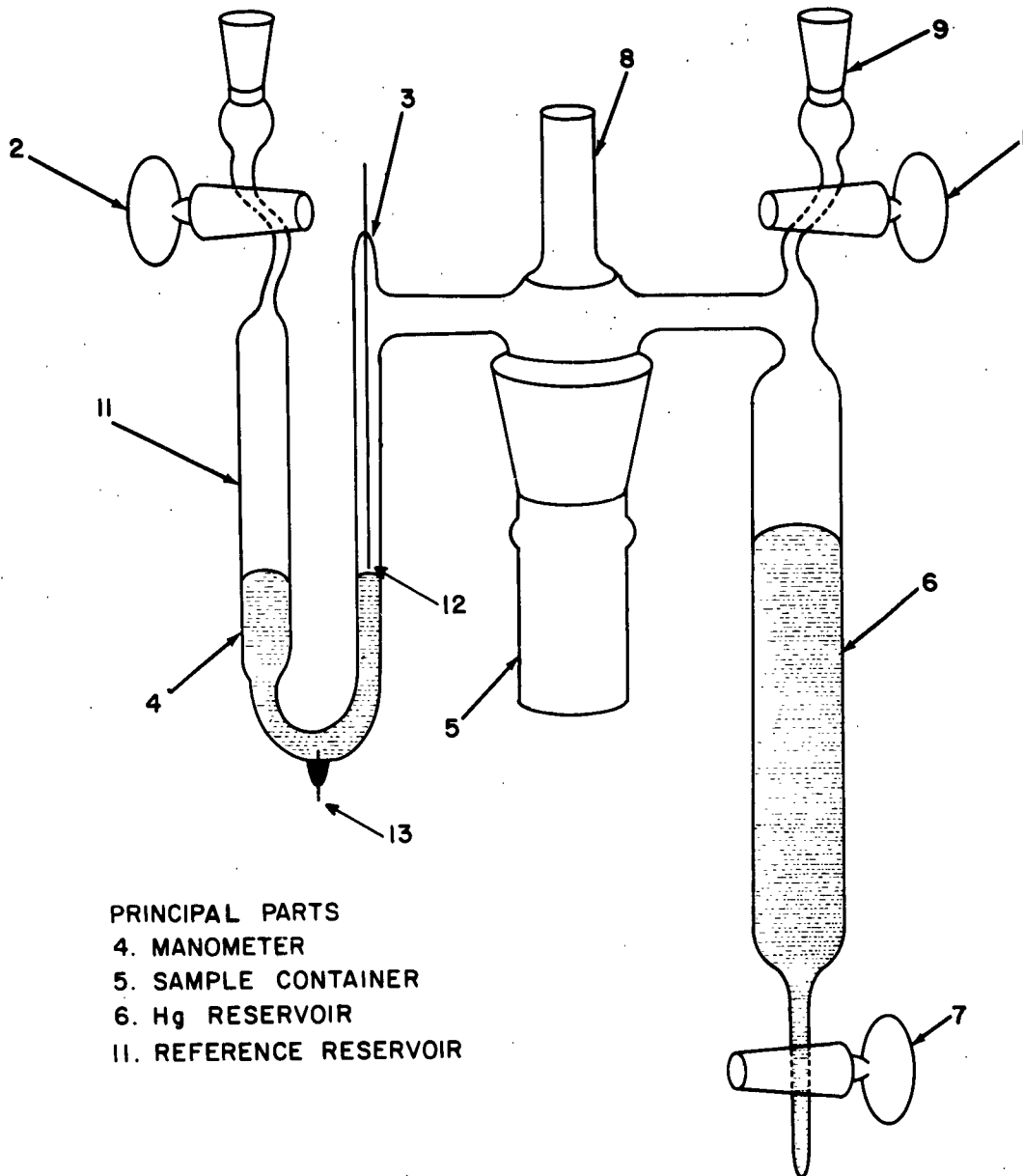
PLANS FOR FUTURE WORK

Further gas evolution studies on the rates of decomposition of silver oxide powders and cathodes in strong KOH electrolyte with and without ZnO present will be carried out. A suitable analytical chemical method for determining the composition of mixtures of Ag, Ag₂O and AgO in the presence of strong KOH electrolyte will be developed. By means of such analysis, coupled with gassing studies, the day-to-day rate of decomposition of AgO in strong KOH electrolyte will be studied.

Further investigations will study the effects of the presence of organic separator material on the rate of decomposition of silver oxide cathode material. A zinc anode will then be added to the system and further decomposition studies carried out both on the cathode and anode. After all the components have been assembled, studies relating charged stand characteristics of silver oxide-zinc secondary batteries to chemical changes in the anode, cathode, separator and electrolyte will be carried out.

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- PRINCIPAL PARTS
4. MANOMETER
5. SAMPLE CONTAINER
6. Hg RESERVOIR
11. REFERENCE RESERVOIR

FIG.1 DIAGRAMMATIC SKETCH OF GASOMETER

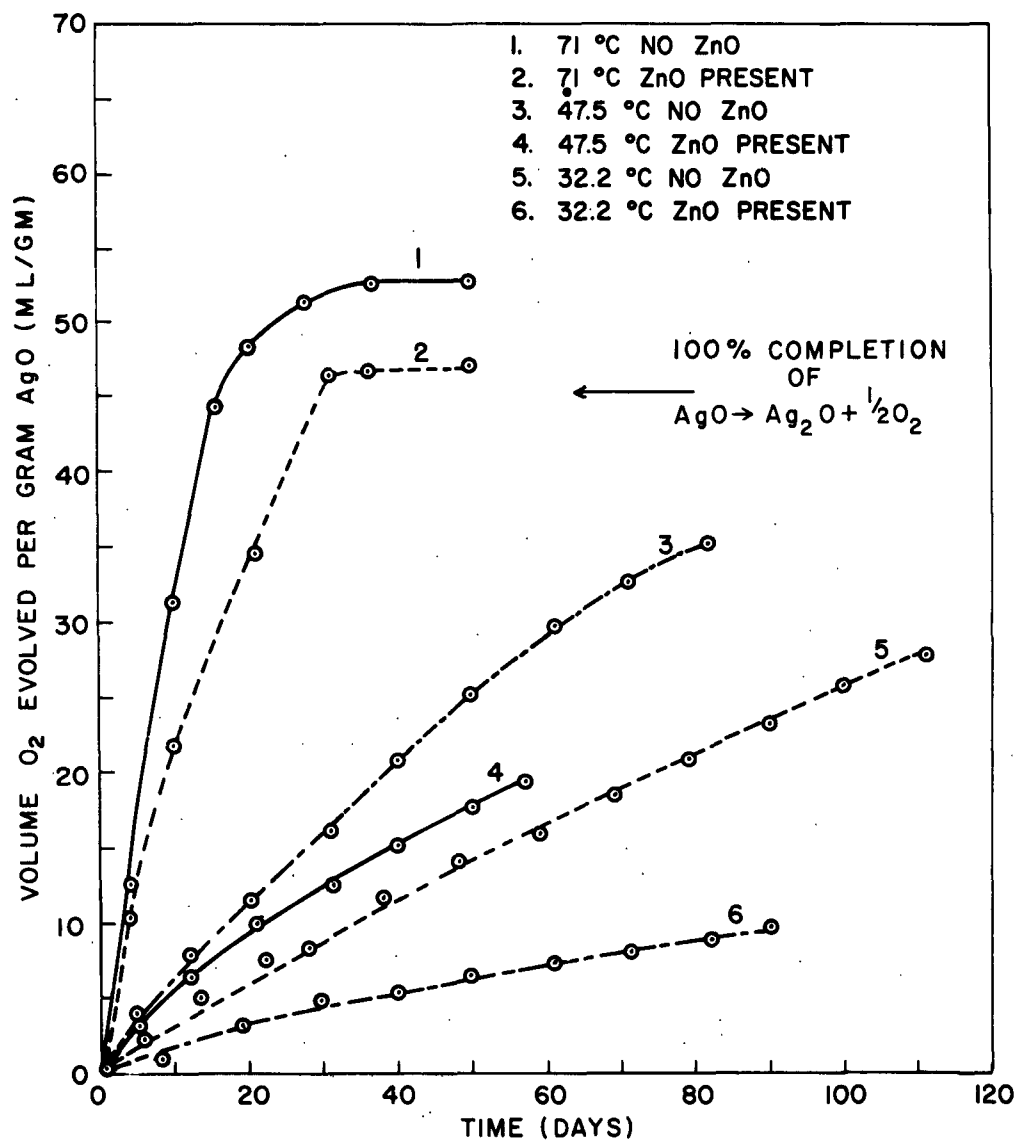


FIG. 2 GASSING RATES OF AgO IN 40% KOH WITH AND WITHOUT ZnO ADDITIVE AT VARIOUS TEMPERATURES

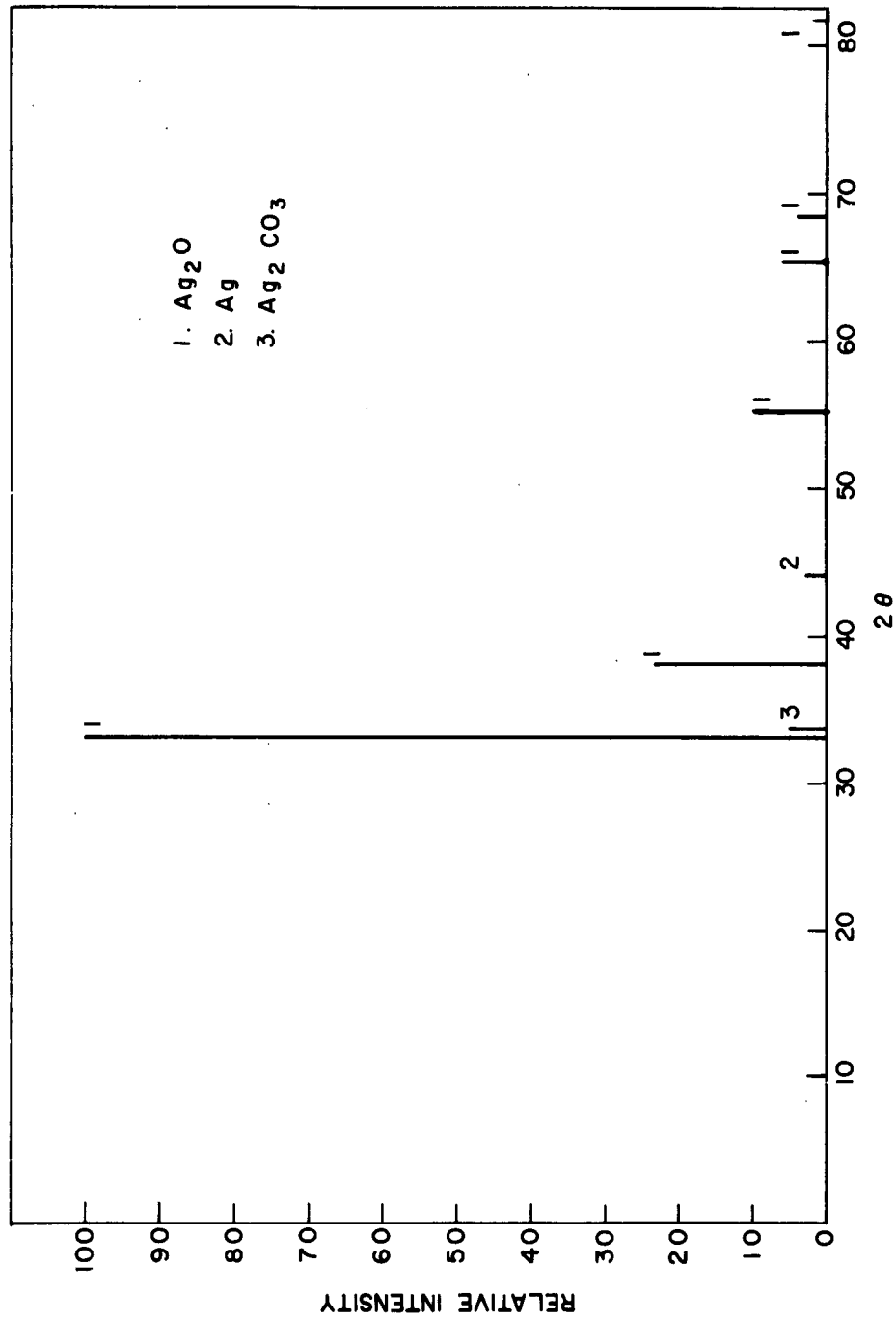
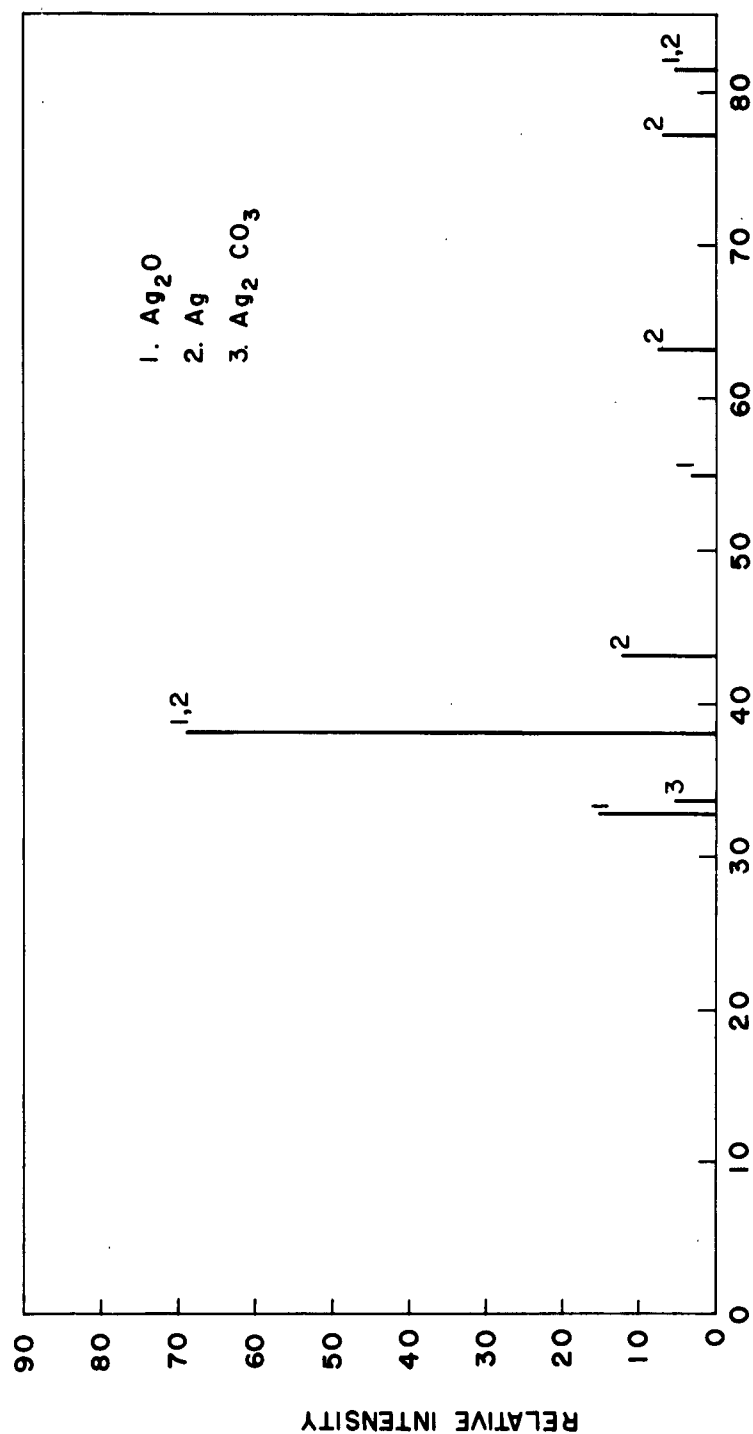


FIG. 3 X-RAY DIFFRACTION PATTERN FOR THE BLACK FRACTION RESULTING FROM TREATMENT OF AgO WITH CONCENTRATED KOH ELECTROLYTE AT 71°C



2θ

FIG. 4 X-RAY DIFFRACTION PATTERN FOR THE WHITE SPONGY FRACTION RESULTING FROM TREATMENT OF Ag_2O WITH CONCENTRATED KOH ELECTROLYTE AT 71°C

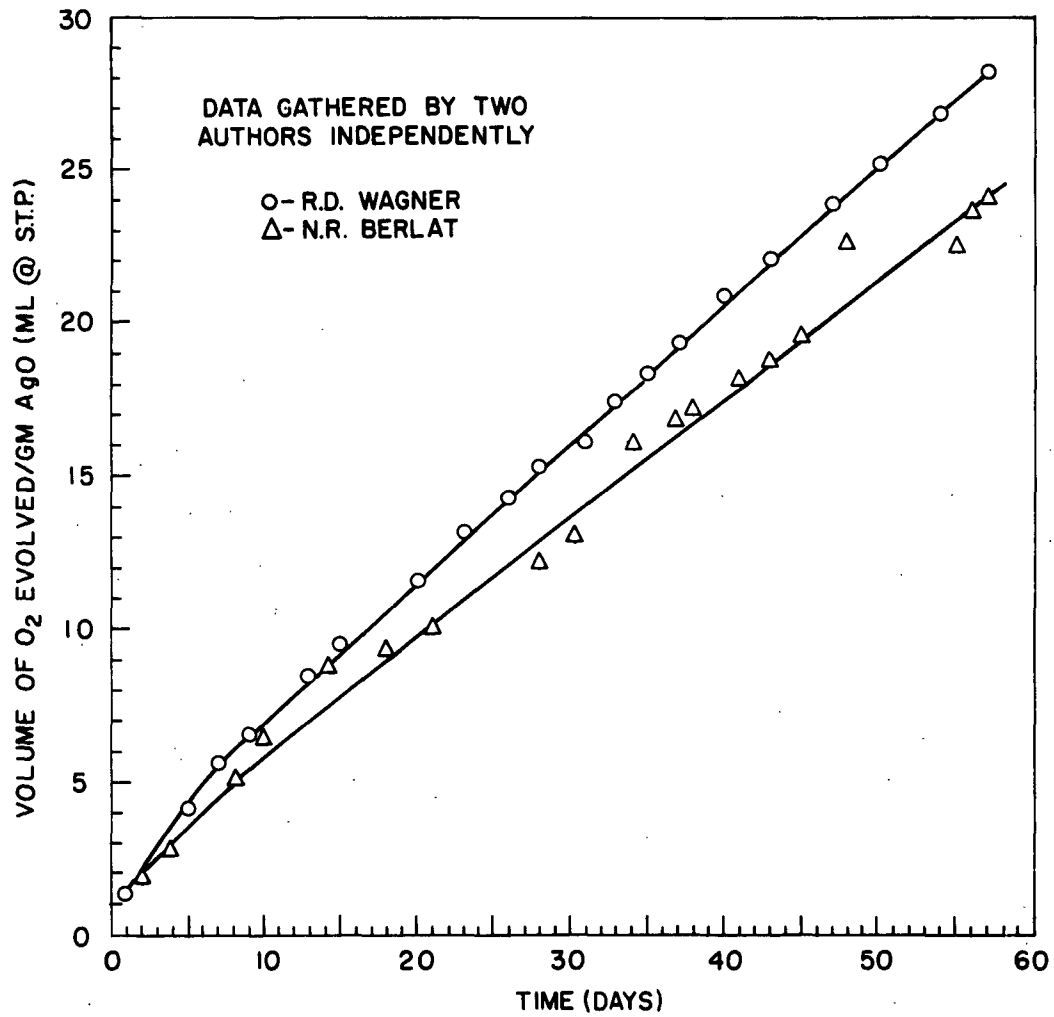


FIG. 5 RATE OF GASSING OF AgO IN CONCENTRATED
KOH ELECTROLYTE AT 47.5 °C

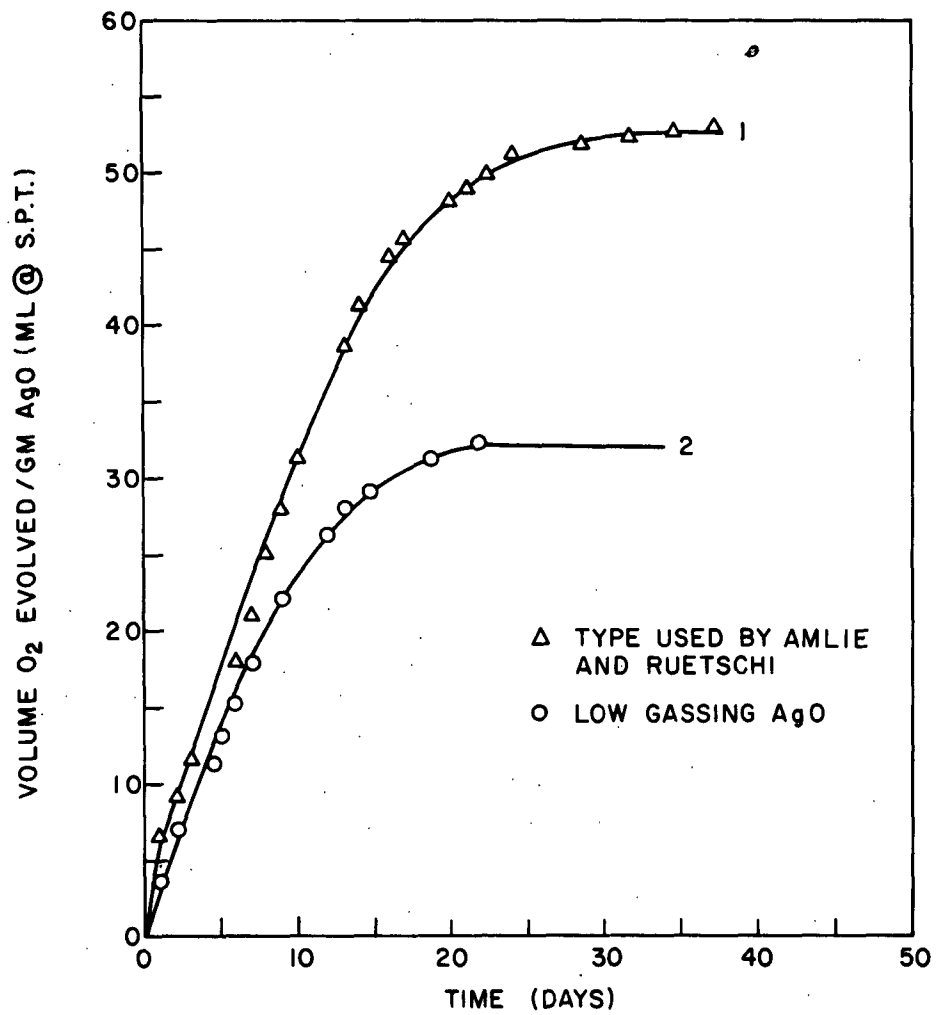


FIG. 6 COMPARISON OF RATE OF GASSING AgO OF THE TYPE USED BY AMLIE AND RUETSCHI WITH RATE OF GASSING OF LOW GASSING AgO AT 71° C

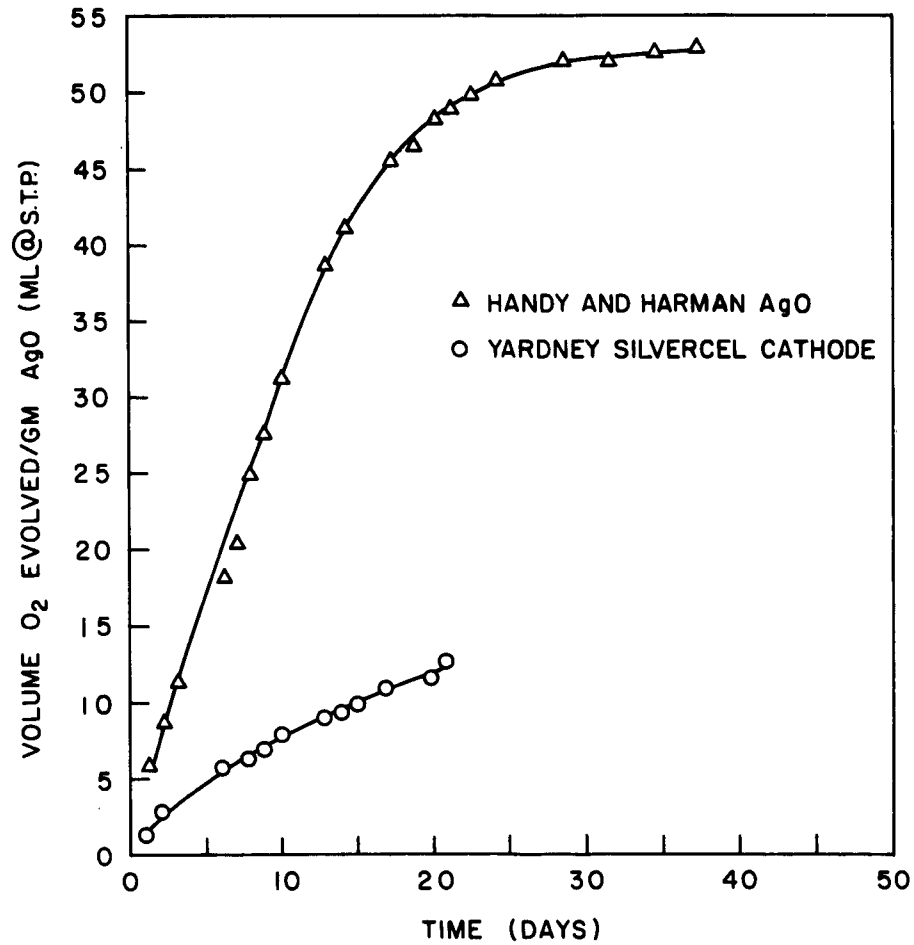


FIG. 7 COMPARISON OF RATE OF GASSING OF HANDY AND HARMAN CHEMICALLY PREPARED AgO WITH THAT OF YARNEY SILVERCEL CATHODE MATERIAL AT 71°C

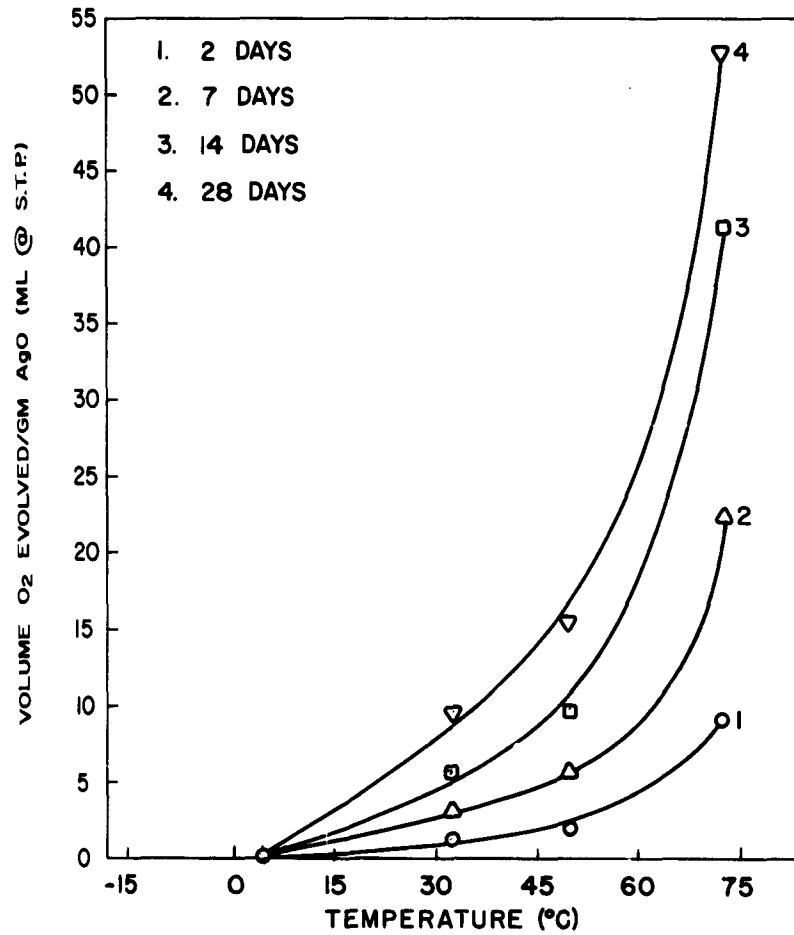


FIG. 8 DEPENDENCE OF RATE OF GASSING UPON TEMPERATURE AT VARIOUS TIMES

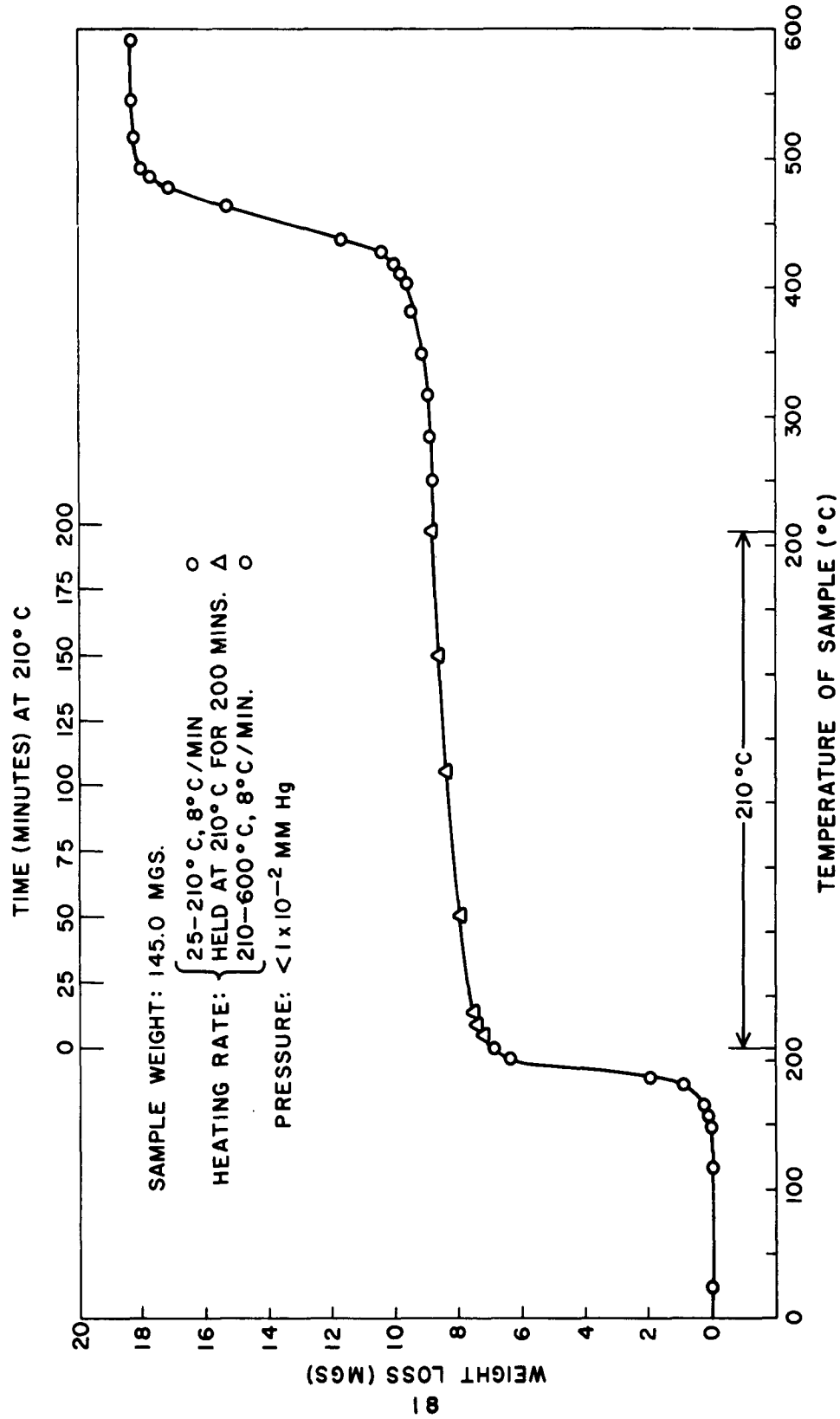


FIG. 9 THERMOGRAVIMETRIC ANALYSIS OF HANDY AND HARMAN AgO

Table I. Constituents of Samples in Gasometers

(In each case about one-half gram of solid in 20 ml of electrolyte was used.)

<u>Temperature</u>	<u>Solution</u>		<u>Solid</u>
	<u>Concentration KOH</u>	<u>Concentration ZnO</u>	
4.4°C (40°F)	40%	0	AgO
32.2°C (90°F)	40%	0	AgO
32.2°C (90°F)	40%	6.5%	AgO
47.5°C (118°F)	40%	0	AgO
47.5°C (118°F)	40%	6.5%	AgO
71.0°C (160°F)	40%	0	AgO
71.0°C (160°F)	40%	8.54%	AgO
71.0°C (160°F)	40%	0	AgO
71.0°C (160°F)	40%	0	(low gassing)* Yardney Silvercel Cathode
71.0°C (160°F)	40%	0	Ag ₂ O
71.0°C (160°F)	40%	8.54%	Ag ₂ O

*The term "low gassing" was used by Handy and Harman on the label of one lot of AgO. Telephone conversations with Handy and Harman concerning the term indicated that there is no difference in chemicals or catalysts used in the two lots but rather process and quality control. Their experimental results show a decrease in gassing.

Table II. Typical Results Obtained in the Crucible Analysis of AgO

Wt. Sample (gms)	Wt. Loss #1 (gms)	Wt. Loss #2 (gms)	% AgO	% Ag ₂ O	Total %	Theoretical	
						Total Wt. Loss (gms)	Total Wt. Loss (gms)
0.4121	0.0224	0.0312	84.178	30.939	115.117	0.0536	0.0532
0.4226	0.0230	0.0323	84.287	31.874	116.161	0.0553	0.0546
0.4988	0.0298	0.0344	92.527	13.360	105.887	0.0642	0.0644
0.4764	0.0292	0.0327	94.928	10.644	105.592	0.0619	0.0615
0.4228	0.0264	0.0282	96.705	6.167	102.872	0.0546	0.0546
0.6515	0.0408	0.0440	96.990	7.116	104.106	0.0848	0.0842

The theoretical total weight loss given in the last column of the table was calculated on the basis of AgO decomposing according to the following single-step reaction: $\text{AgO} \xrightarrow{\Delta} \text{Ag} + \frac{1}{2}\text{O}_2$.

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BIBLIOGRAPHIC INFORMATION

	DESCRIPTORS	CODES	SECURITY CLASSIFICATION AND CODE COUNT	DESCRIPTORS	CODES
SOURCE	NOL technical report	NOLTR		Unclassified - 13	U013
REPORT NUMBER	62-187	620187	CIRCULATION LIMITATION		
REPORT DATE	23 Oct. 1963	1063	CIRCULATION LIMITATION OR BIBLIOGRAPHIC		
			BIBLIOGRAPHIC (SUPPL., VOL., ETC.)		

SUBJECT ANALYSIS OF REPORT

	DESCRIPTORS	CODES	DESCRIPTORS	CODES	DESCRIPTORS	CODES
Silver		SILV	Batteries	BATT		
Oxides		OXID				
Decomposition		DECM				
Potassium		POTA				
Hydroxide		HDRO				
Electrolyte		ELCL				
Temperature		TEMP				
Gas		GASE				
Rates		RATE				
Thermodynamics		THED				
Metals		META				
Chemistry		CHEM				

Naval Ordnance Laboratory, White Oak, Md.
(NOL technical report 62-187)
THE DECOMPOSITION OF ARGENTIC AND ARGENTOUS
OXIDES IN CONCENTRATED KOH ELECTROLYTE (U),
by Frederic M. Bowers and others. 23 Oct.
1962. 20p. diagr., tables. Task RUTO 3E000/
217. UNCLASSIFIED

The rate of decomposition of AgO in 40%
(10N) KOH electrolyte was studied at tempera-
tures ranging from 4.4°C to 71°C. No decomp-
osition was observed at 4.4°C. After 35 days
at 71° there was complete reduction to Ag₂O
and metallic silver. The addition of 6.5 -
8.5% ZnO by weight decreases the rate of
decomposition of AgO and possibly prevents
the decomposition of Ag₂O. The decomposition
of Ag₂O to metallic silver under the condi-
tions studied is thermodynamically feasible.

Abstract card is unclassified.

1. Argentio
oxides -
Decomposition
2. Silver oxide
3. Argentous
oxide -
4. Decomposition
Electrolytes,
Potassium
hydroxide
Batteries,
5. Silver
oxide-zinc
- I. Title
- II. Bowers,
Frederic M.
- III. Project

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